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Elastic properties of packing of granulated cork: Effect of particle size

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ABSTRACT

Cork is a natural raw materials used in numerous traditional and innovative applications among which the most famous are cork stoppers, insulation boards, and wall and floor covering. Cork exhibits a relatively homogeneous honeycomb structure composed of polyhedron cells filled with gas, which gives its specific properties such as elasticity or hydrophobic behavior. Mechanical properties of cork have been studied from a long time. However, in many industrial applications cork is increasingly used as granulate (>200 μm) or powder (<200 μm) obtained by milling processes. By generating failure in the raw material, the comminution of cork could damage cells and induce modifications in the mechanical behavior of cork particles but the relation between the size of the particles and the mechanical properties of the packaging is still little known. In this study, we investigate the effect of cork particles size reduction on the mechanical properties of the packing. To this end, eighteen discrete size fractions of cork from 0 to 25 μm to 2.5–3 mm, generated by impact milling were characterized in term of shape, loaded density, and elastic recovery. Experiments show that impact milling mainly affects the elastic properties of particles less than 200 μm. The mechanical behavior of cork particles was also described by model based on a mixing law taking in account the size and the shape of particles and the amount of damaged cells in the particles. The fitting of the model to the experimental data suggests that the elastic properties of the packing of cork particles have to be related to the content of damage cells more than to their size.

1. Introduction

Cork is a wonderful natural raw material known from ancient time for its low density, hydrophobic behavior, high elastic recovery, sound and electrical insulation. Cork is particularly abundant in the bark of a specific species of oak: *Quercus suber* L., commonly known as cork oak, which is primarily grown in the western Mediterranean. Europe produces 80% of the world's cork and almost three quarter of them in Portugal (Silva et al., 2005). Cork exhibits a relatively homogeneous honeycomb structure composed of polyhedron cells filled with gas which could represents up to 85–90% of the total volume (Pereira, 2015). Each cell is constituted of two hexagonal bases with prismatic faces (Pereira, 2007). Suberin is the major component of cork cell wall and represents 53% in average of the structural components. The other main components are lignin (26%), cellulose and hemicelluloses (≈10%). This specific chemical composition together with honeycomb structure filled with gaz explain its low density, its hydrophobicity, and significantly contribute to its specific mechanical properties (Fortes and Nogueira, 1989; Anjos et al., 2014; García et al., 2015; Pereira, 2015). Cork is used in both traditional and innovative industrial applications among which the most known are cork stoppers for wine industry (Demertzi et al., 2016), insulation boards (Torgovnikov and Vinden, 2006), wall and floor covering (Ferreira et al., 2016), biosorbents (Pintor et al., 2012), adhesive joints (Abenojar et al., 2014) (Barbosa et al., 2015) and some niche markets as shoes soles (Reiber et al., 2002) or floating key ring, etc. In most applications, cork is used in the form of polydispersed cork particles agglomerated with an organic binder and moulded at controlled heat and pressure (Sanchez-Saez et al., 2015a). In these cases, a milling step reduces the raw material into a target range of particles size. The upstream steps of the process remains poorly studied (Armstrong, 1891; Saverio, 1930) and is currently performed based on empirical knowledges. However, a better understanding of the different solutions for cork milling is of great importance to optimize the process chain, ensure a constant quality, reduce the waste and energy consumption (Rives et al., 2012). In addition the mechanical properties of cork strongly depends on the failure path that can bypath or cross the cells depending on the stress concentration and fracture toughness.

For the formulation of cork enriched materials, several authors have studied the influence of cork particles size distribution on

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Nomenclature

List of symbols and abbreviations

a	Specific dimensions of the particle shape
AR	Aspect ratio (shape factor)
C	Circularity (shape factor)
d_c	Diameter of a sphere in which a cork cell is embedded
d_p	Diameter of the circumscribe sphere of the particle.
DS	Direct sieving protocol
ER	Elastic recovery of the powder bed
h_c	Prism height of the cell
H_p, H	Height of the packing bed under confining stress and after unloading
l_c	Base edge of the cell
MS	Milling and sieving protocol
N_t N_b and N_s	Number total of cells in a particle, in the bulk and the surface of a particle
N_d	Number of damaged cells in a cork particle
S	Solidity (shape factor)
SEM	Scanning electron microscope
V_c	Volume of the cell
V_{Sphere}	Volume of the circumscribe sphere to a particle
V_p, V_b, V_s	Volume total, volume of the bulk and volume of the outer layer of cork particle
X	Mechanical properties of a particle bed.
X_d and X_u	Mechanical properties of a bed constituted of damaged and undamaged particles.
W_p	Individual weight of the particles
η and η_s	Content of damaged cell in and at the surface of a particle
ρ_{380kPa}, $\rho_{1130kPa}$	Loaded density at 380 kPa and 1130 kPa
ρ_0	Bulk density of cork particle

the strength and failure of composites (Sanchez-Saez et al., 2015a; Jardin et al., 2015), (Sanchez-Saez et al., 2015b; Fernandes et al., 2014). At high solid fractions of cork particles, the macroscopic behavior of the materials is significantly affected by the physical properties of the particles. It is the case for example for wine stoppers made with agglomerated cork particles (ratio >51%). In this study, we investigate the mechanical behavior of cork particles packing according to their size distribution and the proportion of damaged or undamaged cells. A simple model is proposed to predict the strength properties as a function of the amount of damaged cells. Finally, the influence of the milling step on the properties of cork is discussed.

2. Material and methods

2.1. Cork raw material

Cork particles used in this study are industrial samples used for the production of agglomerated cork stoppers coming from a same industrial batch. In this industrial process, after harvesting, the cork from reproduction plank, was boiled, ground by an impact mill and sieved to eliminate the finest fraction (<300 μm), which mainly corresponds to dust, lenticular filling materials and the friable phloemic material (Pereira, 2007). The particle size distribution ranges between 0.30 and 3.5 mm.

2.2. Dimensions and volume of cork cells

Cork cells can be assimilated to right hexagonal prisms (Fig. 1a) (Fortes and Nogueira, 1989). Hence their volume could be estimated by

$$V_c = \left(\frac{3\sqrt{3}}{2}.{l_c}^2\right).h_c \quad (1)$$

Where h_c is the height of the prism and the l_c is the width of the hexagonal base. The average values h_c and l_c have been determinated from SEM image analysis performed on 350 cork cells with an environmental scanning electron microscope (SEM) (Analytic FEI Quanta FEG 200, FEI, Hillsboro, OR, USA) equipped with a BSED detector (Fig. 1b and c). Images were taken with an acceleration voltage of 15 kV in a pressure of 0.5 Torr, at a working distance of 10 mm. These data were fit with a gaussian distribution from which $l_c = 25.0 \pm 5.5$ μm and $h_c = 44.1 \pm 8.5$ μm have been determinated and the corresponding volume, $V_c = 71, 6.10^3 \pm 14, 4\ \mu m^3$ has been calculated. These values are consistent with the data of the literature which depends on oak tree species, age of the trees and harvesting period (Pereira et al., 1987; Lopes et al., 2001). In the following these measured values are used as cell characteristic dimensions.

2.3. Preparation of monodisperse fractions

Two separation protocols were used to produce fractions at different particle size: direct sieving of raw material (DS) and an additional milling before the sieving step (MS). Eleven coarse fractions were obtained with the DS protocols and referenced as DS followed by the opening of the two consecutive sieves (DS315-450, DS450-560, DS560-710, DS710-800, DS800-900, DS900-1000, DS1000-1250, DS1250-1400, DS1400-2000, DS2000-2500 and DS 2500–3000). For the MS protocol, a 100 UPZ Hosokawa-alpine® (Augsburg, Germany) impact mill was employed at the speed of 12000 rpm with a 300 μm screen. Then an air jet sieving device (200LS-N, Hosokawa Alpine®, Augsburg, Germany) was used to get the finest fractions: MS0-25, MS25-50, MS 50–75,MS 75–100, MS100-150, MS150-200, MS200-315, MS315-450. The 315–450 μm class was obtained with both protocols allowing an evaluation of the discrepancy between the DS and MS values. Because of the broad distribution of particle size considered in this study, different methods were necessary for the characterization of fractions. In the following, all measurements were performed on preliminary dried fractions (48 h at 60 °C).

2.4. Characterization of fractions <450 μm

The median size of fractions <450 μm (MS0-25 to MS 315–450 and DS315-450) were characterized using a laser diffraction particle size analyzer Hydro 2000S (Malvern Instruments Ltd., United Kingdom) with liquid ethanol (ethylic alcohol: 96% v/v) carrier to avoid particle swelling. The determination of the size is based on the assumption that particles are spheres.

2.5. Characterization of fractions >450 μm

2.5.1. Size and shape of particles

Digital image analysis was performed on the coarser fractions (size >450 μm, DS450-560 to DS2500-3000), according to the protocol of Vaezi et al. (2013). Cork particles were dispersed on the overall surface of a scanner. 5 different snapshots at 600 dpi (300–400 particles/image) were acquired per sample. The "analyze particles" plugin of ImageJ software (Abramoff et al., 2004) was used to determine particle size and shape. It is worth noting that, even if the measurements are done in 2D, an equivalent volume is calculated assuming a spherical shape. Three 2D- shape factors were considered:

– Circularity C which is a function of the perimeter P and the area A of the shape: $C = \frac{4*\pi*A}{P^2}$. ($0 < C \leq 1$). A value of 1 indicates a perfect